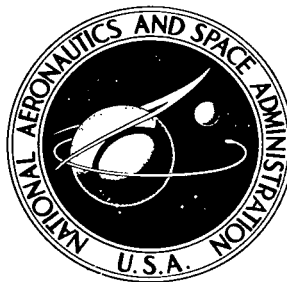


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INFLUENCE OF CHEMISORBED FILMS ON ADHESION AND FRICTION OF CLEAN IRON

by Donald H. Buckley
Lewis Research Center
Cleveland, Ohio



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ABSTRACT

Studies were conducted in a vacuum of 10^{-10} torr with clean polycrystalline iron and with iron containing various films. Results indicate strong adhesive forces for the clean iron; because of the magnitude of these forces friction could not be measured. These forces were appreciably reduced in the presence of adsorbates. Carbon dioxide dissociated on the surface, resulting in metal oxide and carbon monoxide. Water vapor was more effective in reducing friction than was oxygen of the same exposure. With hydrocarbons, the number of carbon-to-carbon bonds influenced friction and adhesion. Friction decreases with chain length and with the number of carbon-to-carbon bonds for a particular chain length.

INFLUENCE OF CHEMISORBED FILMS ON ADHESION AND FRICTION OF CLEAN IRON

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SUMMARY

Adhesion and friction studies were conducted in a vacuum of 10^{-10} torr with polycrystalline iron. Experiments were conducted with a hemispherical rider contacting a flat surface. Polycrystalline iron was examined both in a clean state and in the presence of adsorbed gas films. The gases adsorbed to the surface were oxygen, carbon dioxide, water vapor, ethylene, acetylene, and a homologous series of hydrocarbons of methane through decane. Adsorption and desorption of gases during the experiments were monitored with a mass spectrometer. The applied load on the specimens was negligible in the adhesion experiments. A load of 10 grams was used in the sliding friction experiments.

The results of the investigation indicate that, for clean iron surfaces, adhesive forces are very high even under negligible normal load. Under such conditions, and within the limits of this study, the measure of friction force becomes a practical impossibility. The presence of adsorbed gases appreciably reduced adhesion and made the measurement of friction possible. With oxygen, the greatest reduction in friction occurred with the adsorption of a monolayer. Carbon dioxide dissociated during the course of sliding friction experiments, resulting in metal oxide and carbon monoxide. Water vapor was more effective in reducing friction than either oxygen or carbon dioxide at equivalent concentrations. With hydrocarbons, the number of carbon-to-carbon bonds influenced friction.

INTRODUCTION

The adhesion of clean, unalloyed polycrystalline iron is extremely important to the understanding of the influence of various surface films on adhesion and friction. If a clean surface can be obtained, the effect of selectively adsorbed materials can be quali-

tatively and quantitatively studied. While a clean surface itself is not an end, it provides a point of reference.

For a considerable number of years, efforts have been made to obtain clean metal surfaces. The object of many of these attempts has been to gain a better understanding of the effect of various surface properties of metals on mechanical behavior and to provide for a controlled study of the interaction of these surfaces with various selected media. One of the most difficult surfaces to clean, and at the same time one of the most important, is polycrystalline iron. The difficulty is not the removal of surface films but the continuous diffusion of impurities from within the iron to the surface on heating. The diffusion of carbon to the surface has been held (ref. 1) to be the principal factor responsible for the inability to maintain a clean surface. Cleaning procedures involve some type of heating, and the task of obtaining a clean iron surface has been felt by some to be all but impossible (ref. 1). Other studies, however, indicate that, with care, clean iron surfaces can be obtained (refs. 2 to 5). The most effective way of accomplishing this task appears to be to reduce the initial carbon concentration to a very low level.

The objectives of this investigation were (1) to measure the adhesion and friction of clean polycrystalline iron surfaces and (2) to measure the effect of various adsorbates on the reduction of the adhesion and friction of these surfaces. Simple touch-contact adhesion experiments under negligible load were conducted in vacuum (10^{-10} torr) with triple zone refined iron containing less than 8 ppm of carbon. Sliding friction experiments were conducted with a 0.475-centimeter-radius hemispherical slider in contact with a 2.54-centimeter-diameter flat disk. Friction experiments were made with the following vapors adsorbed to the iron surface: oxygen, carbon dioxide, water vapor, methane, ethane, ethylene, acetylene, propane, butane, hexane, octane, and decane. The sliding speed employed in the friction experiments was 0.001 centimeter per second, and the load was 10 grams.

Initial friction experiments were conducted with polycrystalline iron in contact with the same material. Later experiments were conducted with an aluminum oxide hemispherical rider in contact with flat polycrystalline iron disks. The aluminum oxide rider was selected to facilitate the detection of subtle changes in friction with various adsorbed films. With the iron slider, these subtle changes could not be detected because of the marked stick-slip motion and high friction values.

MATERIALS

The iron disk and rider specimens used in this study were electron-beam float zone refined. They contained less than 8 ppm of carbon. The gases were all high-purity reagent grade. The fluids hexane, octane, and decane were 99.99, 99.85, and 99.49 mole

percent, respectively. The commercial high-purity hydrogen gas used in this study was further purified (of possible water vapor, hydrocarbons, etc.) by passing it through a liquid-nitrogen-cooled, molecular sieve.

All specimens were cleaned on abrasive paper after they had been machined to shape. They were next electropolished in phosphoric acid. The specimens were then sealed in evacuated quartz ampules and held at a temperature of 800° C for a week. The specimens were subsequently re-electropolished.

APPARATUS

The apparatus used in this investigation is shown schematically in figure 1. The specimens were a 2.54-centimeter-diameter flat disk and 0.475-centimeter-radius hemispherical rider specimen (shown in insert of fig. 1). The disk specimen was mounted on

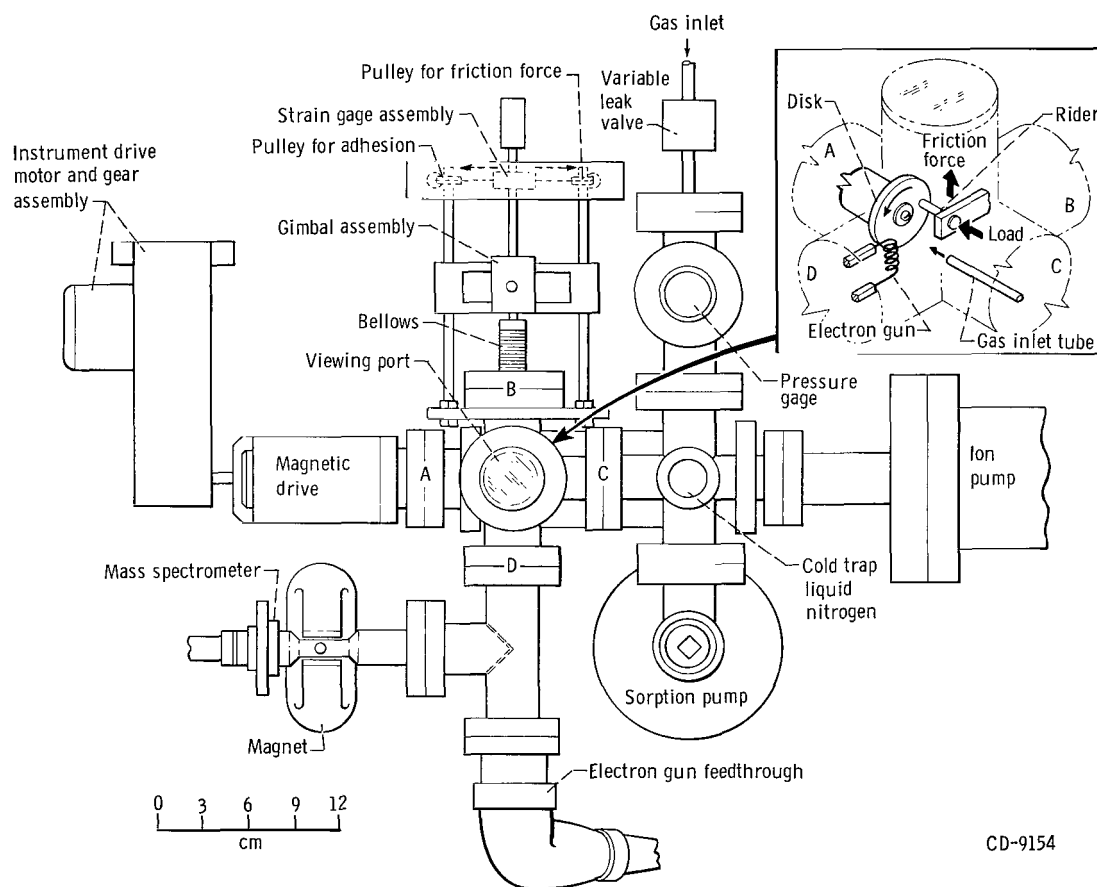


Figure 1. - Vacuum friction apparatus.

a shaft which was magnetically driven by a motor and gear assembly. The linear sliding velocity employed in these experiments was 0.001 centimeter per second.

The rider specimen was mounted in an arm which was gimbal mounted and bellows sealed to the vacuum-chamber wall. The rider specimen was loaded against the disk surface with dead-weight loading. Perpendicular to the loading device was a strain gage for monitoring friction force. The measurement of adhesive forces involved the application of a force opposite to the direction of the load to separate the parts. Breakaway forces were measured by filling a polyurethane bucket with liquid gallium. After the specimens separated, the gallium bucket was weighed and the breakaway load was thus determined.

Cleaning of specimen surfaces in the vacuum chamber was achieved with an electron gun, and the gaseous species in the vacuum chamber were monitored with a mass spectrometer.

The vacuum system was rough pumped to 10^{-3} torr with a sorption pump, and pressures to 10^{-10} torr were achieved with an ion pump. A grid was provided in the pump throat to eliminate ionization in the specimen region. A liquid-nitrogen trap was used for cryopumping. Pressure was measured by a cold-cathode ionization gage, as well as by the pump current gage.

Gases were introduced into the vacuum chamber through a variable-leak valve. A separate vacuum sorption pump was used to evacuate the gas line connecting the cylinder of gas to the variable-leak valve. Both the gas line and the vacuum system were baked out with heating tapes and infrared lamps.

EXPERIMENTAL PROCEDURE

Specimen Cleaning

A number of techniques are used to obtain clean surfaces in a vacuum. Two of the more commonly used are the ion-bombardment and the electron-beam techniques. Based on friction results obtained in an earlier study with tungsten, the electron-beam cleaning of surfaces was selected for use in this investigation (ref. 6).

The iron surfaces used in this study were cleaned by electron bombardment in the vacuum chamber when the pressure reached 10^{-10} torr. The specimens were heated with the electron gun to a bulk temperature of 800°C (surface temperatures were considerably higher), and hydrogen gas was admitted to the chamber to reduce surface oxides. Once the surface oxides were reduced (as determined with the mass spectrometer by monitoring the water-vapor peak), the temperature was increased to 1000°C , and the iron surface was electron bombarded for 3 hours to remove the adsorbed hydrogen.

Gas Adsorption

The specimens were cooled to room temperature after the electron bombardment, and the gas was admitted to the specimen zone through a variable-leak valve and a tube. The gases were charged from their cylinders into a line outside the vacuum chamber. Prior to the admission of gas, the line was thoroughly evacuated with a sorption pump and was baked out. The evacuated supply line was then purged three times by alternately filling and evacuating it. Gas was bled into the chamber for adsorption studies to some pressure above 10^{-10} torr and held at that pressure for a specific period of time in order to obtain the proper exposure in torr-seconds. Only for the most prolonged exposure of 700 torr-seconds was the gas bled in at a pressure greater than 10^{-4} torr.

Mass spectrometer traces were obtained during gas admission to the specimen surface, during the friction experiment, and following the experiment. Background data for the mass spectrometer were obtained with a saturated specimen surface at room temperature, that is, when the specimen surface was completely covered with a monolayer.

RESULTS AND DISCUSSION

Iron Sliding on Iron

The adhesive forces between clean iron rider and disk specimens were measured in a vacuum of 10^{-10} torr after surface cleaning by hydrogen reduction and electron-beam heating. Adhesion of the surfaces occurred on simple touch contact with negligible load. At 20° C a force of 27 grams was required to separate the specimens after they had been in contact for a period of 10 seconds.

The force of adhesion of iron to iron was temperature dependent, as shown in the data of figure 2(a). Increasing the temperature of the specimen surfaces from 20° to 500° C, for a 10-second contact time, resulted in nearly a fourfold increase in the adhesion force.

In addition to the influence of temperature on adhesion, contact time was also noted to affect adhesion. The effect of contact time on adhesion of iron to iron is shown in figure 2(b). At contact times up to 200 seconds the increase in adhesive force with time is very marked. Beyond 200 seconds the increase in adhesive force with contact time becomes less notable. These results are similar to results obtained with copper (ref. 7), where a time dependency was also found for the measured adhesive forces.

The junction between iron specimens when placed into contact under a negligible load at 20° C did not shear at tangential forces up to 600 grams, the limiting measurable force of the apparatus. This increase in force is due to an increase in the contact area

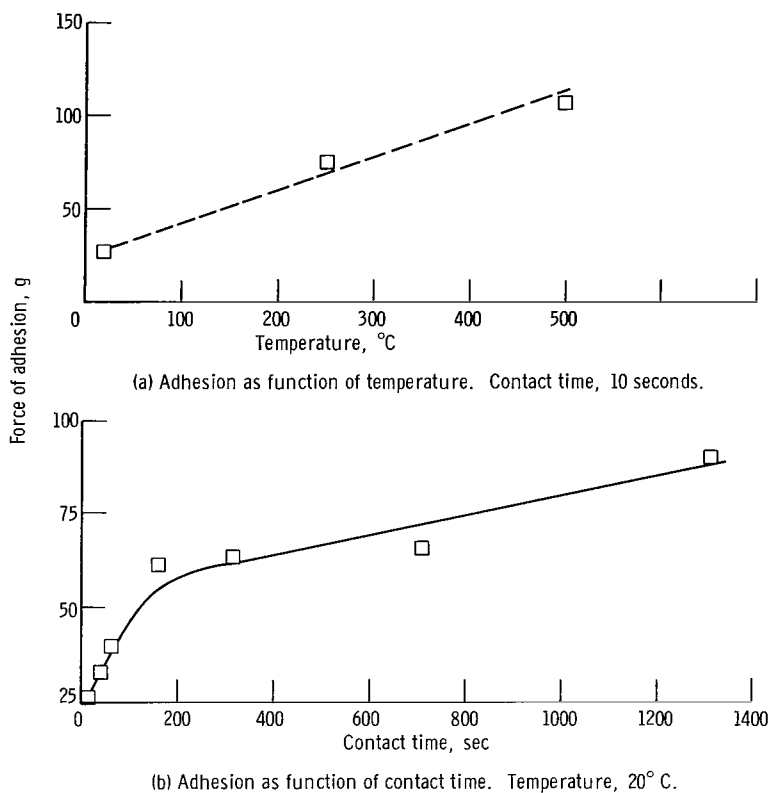
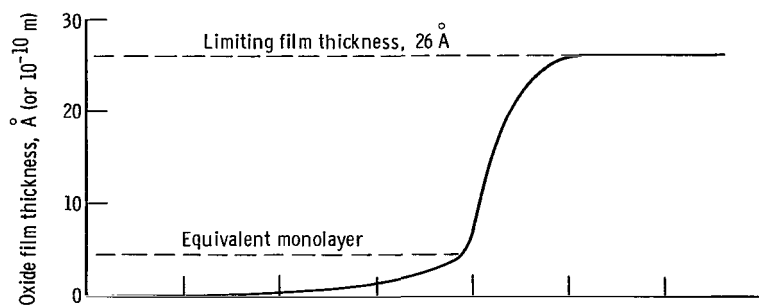


Figure 2. - Adhesion of polycrystalline iron to the same material in vacuum.
Negligible load; pressure, 10^{-10} torr.

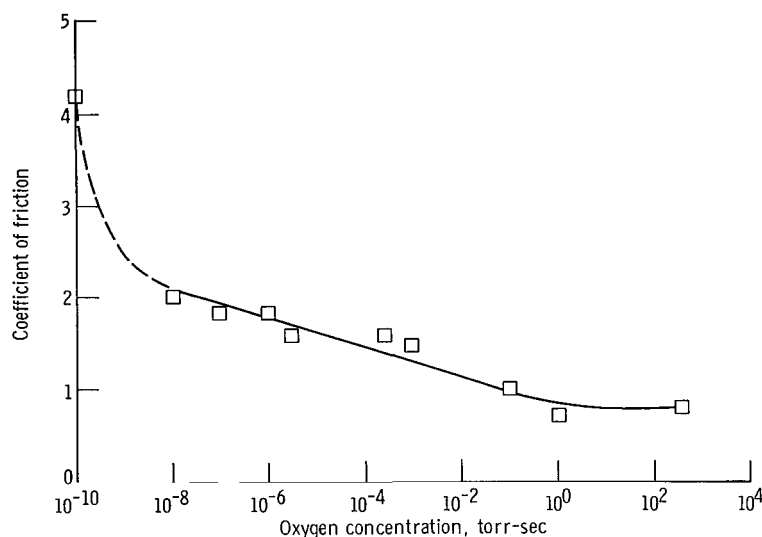
with the application of the tangential force. This result might be predicted from the junction growth theory. Thus, under these conditions it was impossible to measure what would normally be termed a friction coefficient. Attempts to measure the force necessary to move the specimens tangentially becomes, in essence, a shear experiment where the area in shear is continuously increasing because of plastic flow. The limit of this condition is achieved when the real interfacial contact area and the apparent contact area, less voids, become essentially the same.

From the data presented in figure 2 and the description of attempted tangential motion shear force measurement it is obvious that clean iron will adhere very readily to clean iron. The next question relative to such surfaces is what effect the presence of various adsorbed or reacted films have on the clean surface and on the ability of iron to self-weld.

The interaction of oxygen with iron is of most concern in lubrication studies because in most situations oxides of iron are present, usually as multilayer films. The effect of a single monolayer or partial monolayers, as well as thin oxide films, on the adhesion and friction of iron is of interest.



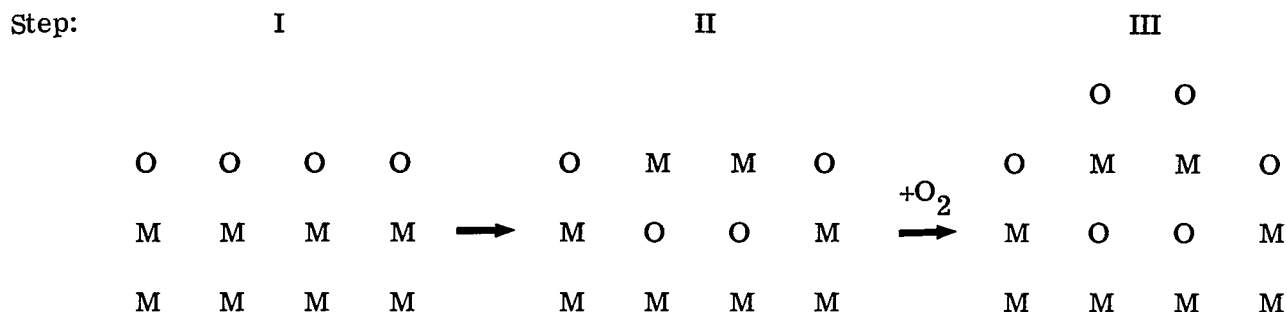
(a) Oxide film thickness (ref. 3).



(b) Coefficient of friction.

Figure 3. - Oxide film thickness and coefficient of friction as functions of oxygen concentration for aluminum oxide sliding on polycrystalline iron. Sliding velocity, 0.001 centimeter per second; load, 10 grams; ambient pressure, 10^{-10} torr.

Some experiments were conducted in which the oxygen was supplied to the clean iron surface at a fixed exposure of 3.0 torr-seconds of oxygen, which is sufficient to form a 26-Å-thick film (2.6×10^{-6} mm) (fig. 3) on the iron surface (ref. 3). This exposure is not critical since this is the limiting film thickness, according to reference 3, for oxygen on iron under these conditions. The admitted oxygen does not adsorb as a uniform film over the entire surface. This is particularly true for a polycrystalline material where certain orientations and grain boundaries are more chemically active than others. Even on a single crystal surface, the presence of imperfections and other defects will result in variations in surface activity. The initiation of the oxidation of iron may be shown by the following sequence proposed in reference 8.



Step I is the formation of an oxygen monolayer, where the surface is ideal. Step II is a position interchange of oxygen with metal. Step III, with the addition of more oxygen, is the formation of the complex surface of mixed metal and oxygen ions. Thus, for iron, more than a simple monolayer of oxygen may be accommodated on the surface in the form of a chemically bonded film.

After the admission of 3.0 torr-second of oxygen to the iron surface (26-Å-thick film) the adhesive forces on the application of negligible loads were also negligible. Further, with a 10-gram load, tangential sliding could be achieved, but a very high friction force was recorded, as shown in table I. The sliding was characterized by a marked

TABLE I. - INFLUENCE OF ADSORBED GASES ON FRICTION
OF POLYCRYSTALLINE IRON IN SLIDING CONTACT
WITH ITSELF IN VACUUM

Gas adsorbed (a)	Coefficient of friction (b)	Principal gases liberated during sliding
None	Complete seizure	None detected
Oxygen (O ₂)	4.66	No oxygen detected
Methane (CH ₄)	8.0	C ₂ H ₆
Ethane (C ₂ H ₆)	8.0	C ₂ H ₆ , CH ₄ , C ₂ H ₅
Ethylene (C ₂ H ₄)	7.0	C ₂ H ₆
Acetylene (C ₂ H ₂)	6.0	C ₂ H ₆

^aGas exposure: 3.0 torr-sec of O₂ at 20° C; 3.0 torr-sec of CH₄, C₂H₆, C₂H₄, and C₂H₂ at 100° C.

^bSliding velocity, 0.001 cm/sec; load, 10 g; ambient pressure, 10⁻¹⁰ torr.

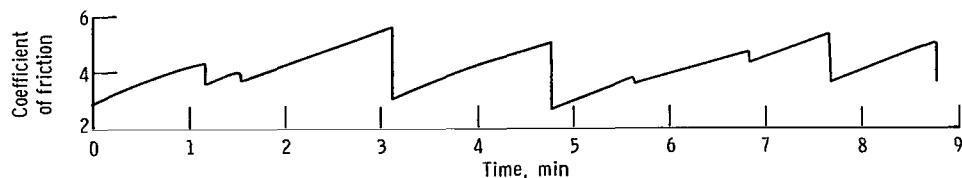


Figure 4. - Coefficient of friction for polycrystalline iron sliding on the same material with an adsorbed layer of oxygen. Sliding velocity, 0.001 centimeter per second; load, 10 grams; ambient pressure, 10^{-10} torr.

stick-slip type motion, as shown in figure 4. The stick-slip motion would indicate that metal-to-metal contact was occurring through the oxide film. This was substantiated on examination of the slider surface after the experiment, since there was evidence of metal transfer and pullout. A mass spectrometer was used to monitor the gases liberated during the sliding experiment, and no evidence of oxygen was detected.

Various hydrocarbons were next adsorbed to the clean iron surface to determine their effect on the adhesion and friction of iron. Because of the high activation energies for the adsorption of hydrocarbons to iron (refs. 8 to 10), the metal surface was held at 100°C during the adsorption process.

Sliding friction experiments could again be conducted with the surfaces in contact with negligible adhesion on simple touch contact. The results obtained in sliding friction experiments are presented in table I. The friction coefficients obtained with all four hydrocarbons were higher than those obtained with oxygen. Further, for the series ethane, ethylene, and acetylene (single, double, and triple carbon-to-carbon bonds), the friction decreased with an increase in the number of carbon-to-carbon bonds. Mass spectrometer data obtained during the course of the sliding experiments revealed the liberation of principally ethane. This would indicate self-hydrogenation of the hydrocarbons. A decrease in friction coefficient with the gases ethane, ethylene, and acetylene has been observed with various atomic planes of tungsten in reference 6. Further, the same type of self-hydrogenation was observed in reference 6 and in the simple desorption studies of reference 8.

Aluminum Oxide Sliding on Iron

With polycrystalline iron sliding on the same material in the presence of a chemisorbed layer, the film is readily penetrated by asperities, with adhesion and extensive disruption of surface and film. This is noticeable even where the film thickness is greater than a simple monolayer, as is the case for oxygen on iron (see the marked stick-slip motion of fig. 4). In order to reduce this marked stick-slip effect and the

disruption of the surface film, an aluminum oxide rider specimen was substituted for iron. Aluminum oxide will adhere to clean iron by an oxygen-iron bonding mechanism (ref. 11), but the massive surface disturbance noted with iron in contact with iron is appreciably reduced.

Sliding friction experiments were, therefore, conducted with aluminum oxide sliding on polycrystalline iron in the presence of various concentrations of adsorbed oxygen. The data of figure 4 show that the greatest reduction in friction is obtained for an equivalent monolayer of oxide. Where film buildup is the greatest (beyond a monolayer), the reduction of friction is the least. Friction is reduced from 1.2 to 0.8 at changes in gas concentration from 10^{-2} to 10^0 torr-seconds, while during monolayer formation the reduction is from 4.2 to 1.2.

Studies were also conducted with the adsorption of carbon dioxide on a clean polycrystalline iron surface. The results obtained in these studies are presented in figure 5. With the adsorption of carbon dioxide the liberation of carbon monoxide was noted during adsorption and during sliding friction experiments. The friction data points for carbon dioxide are compared with the oxygen curve from figure 3. The agreement of the carbon dioxide data points with the oxygen curve and the liberation of carbon monoxide as detected by the mass spectrometer indicate that oxygen is causing the reduction in friction.

Lubricated and unlubricated surfaces alike frequently encounter water as part of the environment. Therefore, the effect of water on the friction characteristic of a clean iron surface is of interest. The effect of water on an iron surface can be seen in figure 5.

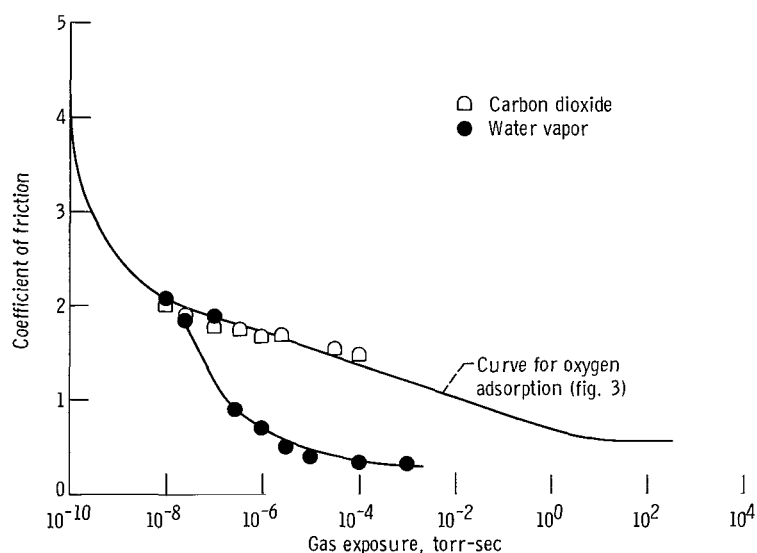


Figure 5. - Coefficient of friction for aluminum oxide sliding on polycrystalline iron in the presence of various concentrations of adsorbed carbon dioxide and water vapor. Sliding velocity, 0.001 centimeter per second; load, 10 grams; ambient pressure, 10^{-10} torr.

Very low concentrations of water vapor resulted in a considerable reduction in friction coefficient. A comparison of the data obtained for oxygen at various concentrations with those obtained for water vapor shows that water vapor is more effective in the reduction of the friction of iron. Both gases adsorb to the iron surface. However, in the adsorption process the oxygen molecule first strikes the surface and then moves about the surface to a site where dissociation into atomic oxygen can take place. The energy of dissociation is about 58 680 calories per mole (245.87 kJ/mole) at 25° C. Subsequent to dissociation, the oxygen will chemisorb. Molecular oxygen will not stick to the surface if it does not find a site for dissociation (ref. 12). Water vapor requires less energy for the dissociation into hydrogen and hydroxyl groups (57 000 cal/mole (238.83 kJ/mole) at 25° C). A further consideration is that water vapor will adsorb directly on the surface without the need for dissociation and site searching, because of the strong dipole moment involved between the water vapor and a clean metal surface (ref. 13). Thus, positively charged sites in the metal will serve to strongly attract the water vapor to the metal surface.

Because of the interest of hydrocarbons in the field of lubrication, some studies were conducted with a homologous series of aliphatic hydrocarbons, methane through decane, adsorbed to iron. The adsorption of hydrocarbons to clean iron involves an activation energy. If the surface of the iron is heated to 100° C, adsorption of the hydrocarbon to iron will occur with dissociation of hydrogen from carbon and with carbon to metal bonding (refs. 9, 10, and 14). Friction data obtained are presented in figure 6. This figure shows that friction decreases with increasing chain length as the number of carbon atoms in the chain length increases from 2 to 8.

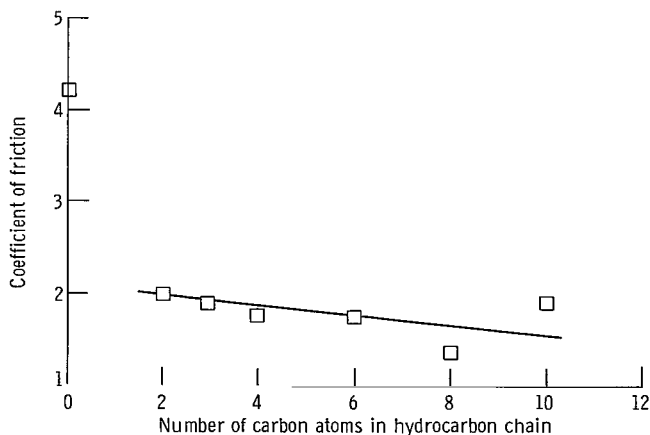


Figure 6. - Influence of the number of carbon atoms in hydrocarbon chain length on friction coefficient of aluminum oxide sliding on polycrystalline iron. Sliding velocity, 0.001 centimeter per second; load, 10 grams; ambient pressure, 10^{-10} torr.

TABLE II. - INFLUENCE OF THE NUMBER OF CARBON-TO-CARBON
BONDS IN AN ADSORBED GAS ON FRICTION COEFFICIENT OF
ALUMINUM OXIDE SLIDING ON IRON

Gas adsorbed to iron surface at 100° C	Number of carbon bonds	Coefficient of friction (a)
Ethane ($\text{H}_3\text{C} - \text{CH}_3$)	1	2.0
Ethylene ($\text{H}_2\text{C} = \text{CH}_2$)	2	1.7
Acetylene ($\text{HC} \equiv \text{CH}$)	3	1.3

^aSliding velocity, 0.001 cm/sec; load, 10 g; ambient pressure, 10^{-10} torr.

The effect of bond type (single, double, or triple) in the C_2 type of carbon-to-carbon bonds on the friction of iron was studied with the aluminum oxide rider in contact with polycrystalline iron, and the results obtained are presented in table II. This table shows that the friction coefficient decreases with increasing number of carbon-to-carbon bonds. The data of tables I and II are in agreement with sliding friction results obtained for tungsten in reference 6. It would be of interest to know whether the carbon chain of 2 atoms is oriented normal to or parallel to the surface. If it is oriented normal to the surface, it is difficult to understand how the number of bonds would affect the friction. Since the distance between carbon atoms is greatest in ethane, less in ethylene, and least in acetylene (ref. 14), results just the opposite of those obtained might be expected. If the chain is flat on the surface, a greater concentration of carbon in the first surface layer is possible with acetylene because of bond lengths.

SUMMARY OF RESULTS

This investigation was conducted to determine the adhesion and friction of polycrystalline iron in contact with the same material or with aluminum oxide.

The following results were obtained for iron sliding on iron:

1. Adhesion of polycrystalline iron to the same material occurred on simple touch contact with the application of negligible load when the iron was cleaned in vacuum.
2. When a tangential force up to 600 grams was applied to the adhered specimens, there was no evidence of fracture of the adhered interface.
3. Both temperature and contact time had an influence on adhesive forces. The higher the temperature or the longer the contact time, the higher the adhesive forces.

4. The adsorption of various gases on iron had a marked effect on adhesion and friction. While the adsorption of oxygen or hydrocarbons reduced adhesion and permitted the measurement of friction, the greater effect was observed with oxygen.

The following results were obtained for aluminum oxide sliding on iron:

1. The adsorption of oxygen, water vapor, and carbon dioxide to iron all effectively reduced friction. The data obtained for various exposures of oxygen and carbon dioxide could be plotted on the same curve. The friction data, coupled with mass spectrometer data indicating a liberation of carbon monoxide, seem to indicate that the protective surface film with the adsorption of either of the gases was iron oxide. Water vapor deviated from this curve and indicated a much sharper reduction in friction coefficient at concentrations equivalent to that of the oxygen and carbon dioxide.

2. Hydrocarbon chain length influenced friction coefficient. A reduction in friction was observed up to a chain length of 8 carbon atoms.

3. The number of carbon-to-carbon bonds in the 2-carbon-atom hydrocarbon influenced friction; the higher number of bonds showed lower friction coefficients.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, June 10, 1968,
129-03-13-02-22.

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